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SANITIZED VERSION OF WORKS LABORATORY ANNUAL QUALITY CONTROL REPORT - 1951 (MARCH 25, 1952)

(SANITIZED VERSION OF CRD DOCUMENT #KLI-1354)

Compiled by
S. G. Thornton
Environmental Management Division
OAK RIDGE K-25 SITE
for the Health Studies Agreement

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WORKS LABORATORY ANNUAL QUALITY CONTROL REPORT - 1951

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Title: WORKS LABORATORY ANNUAL QUALITY CONTROL REPORT - 1951

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WORKS LABORATORY ANNUAL QUALITY CONTROL REPORT - 1951

INTRODUCTION

This report summarizes the Works Laboratory quality control program for 1951. The report presents, first, in a condensed form, an objective view of the scope of quality control of analytical results in the Works Laboratory, and second, lists the average precisions and accuracies of the analyses carried out in 1951. This summary is expected to be of use to plant and laboratory personnel in the planning of analytical programs and in computations concerning uranium accountability.

A brief discussion of the Works Laboratory quality control policy and program, a description of the services performed by the Works Laboratory, some facts concerning the basis of the quality control standards, and lists of the average precisions and biases of analytical methods as computed from control data are included in this report.

THE WORKS LABORATORY QUALITY CONTROL PROGRAM

The quality control program is set up to maintain a constant check on the precisions and accuracies of analyses done in the Works Laboratory. Approximately one tenth of the samples analyzed are control samples. The character of these samples is changed from time to time in order to conform to the changes in the types of samples received in the laboratory.

The responsibility for the quality control program rests primarily within the sections. Each section head maintains a close observation of the precisions and accuracies of analyses done within his section, and is thus able to take necessary corrective measures at the source of difficulty without loss of time. The program is coordinated, however, from the department office. The results of the monthly control program are summarized in reports written by the section heads, and combined into a single report by the department office.

BIASES OF QUALITY CONTROL STANDARDS

The primary basis for all the chemical uranium standards used in Works Laboratory quality control is U_3O_8, the uranium content of which is known to ± 0.02 per cent. This is a greater accuracy than that required for any sample submitted for analysis.

Isotopic uranium standards are based indirectly on precise measurements of product level material by the mass spectrometer. The alpha counters are calibrated on standards of enriched material, and the fission counters are based on the isotopic values of natural uranium and of K-25 product.

The standards used in non-uranium chemical analyses are either chemically pure reagents or in some cases, are standards issued by the National Bureau of Standards or the American Society for Testing Materials.

The permeability and slope factor controls for barrier tubes are standard orifices for the testing machines.

GENERAL SUMMARY OF TYPES OF SERVICES

The following types of services are offered by the Works Laboratory:

Chemical uranium analysis of feed, waste, product, and inventory batches for the operation of the plant and the uranium accountability program.

Isotopic uranium analysis by counting and spectrometer methods for uranium accountability purposes and as a supplement to chemical uranium analyses for plant operations.

Beta and gamma counting determinations on special radioactive materials.

Specification analyses of fuels, lubricants, paints and other materials needed for the plant.

Industrial hygiene analysis of chemical and radioactive contaminants in air, water, river mud, and biological materials for the protection of plant personnel.

Miscellaneous analyses of a wide variety of materials for plant and laboratory research groups.

Representative sampling and transfer of uranium hexafluoride from different containers without changing the isotopic concentration of the sample.

The construction and maintenance of many types of electronic laboratory equipment.

Trouble shooting and consulting services for plant and laboratory groups.

(1) Pickens, M. N., and A. B. Meservey, "Works Laboratory Quality Control Report 27", Carbide and Carbon Chemicals Company. KLI-652, Part 11. May 10, 1951.

NOTES ON THE QUALITY CONTROL PROGRAM IN 1951

As can be seen by a comparison with the annual report for 1950, the precisions and accuracies of several types of analyses done in the Works Laboratory have been improved in 1951. This has been due in part to better methods of analysis. More uniform and stable standards in some cases also contributed to improved precision. Several analyses showed slightly worse precisions in 1951 than in 1950, but the change was usually not significant.

In general, the types of control samples analyzed were the same in 1951 as in 1950, and are not described again in detail (2).

PRECISIONS AND BIASES OF ANALYSES DONE IN THE WORKS LABORATORY DURING 1951

Table I lists the precision and accuracy for each analysis for which a routine control is maintained. The precision, unless otherwise noted, is reported as a limit of error (LE) for one analysis, expressed as a per cent of the mean. This limit of error is the interval which will include 95% of the sample results. The bias is the average difference between the sample mean and the theoretical mean, if any, and is expressed as a per cent of the theoretical mean. The LE of the bias is that for one analysis. If the bias of the control mean is not known, it is usually expressed as being less than some estimated figure.

(2) Pickens, M. N., and A. B. Meservey, "Works Laboratory Annual Quality Control Report - 1950", Carbide and Carbon Chemicals Company, KLI-900. April 16, 1951.

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TABLE I

SUMMARY OF ANALYTICAL CONTROL DATA FOR 1951

	Analysis	Requested % LE_X	Attained % LE_X	% Bias
I. Control Analyses for Uranium Accountability				
A. Chemical Analyses				
1. UF_6				
a) Feed, Waste, Product	% U	± 0.14	± 0.14	0.01 ± 0.04
b) Recovered	% U	± 1.0	± 2.6	-0.2 ± 0.5
2. UF_4	% U	Best obtainable	± 0.29	< 0.3, est.
3. Miscellaneous Impure Samples				
a) Alumina	g U/g	± 3.5	± 3.2	< 2.0, est.
b) Carbon	g U/g	± 3.5	± 3.2	< 2.0, est.
c) Impure U_3O_8	g U/g	± 3.5	± 3.5	-0.9 ± 1.3
d) Water Media				
1) 0.07 g U/l	Total U	± 10	± 11	-1.7 ± 3.1
2) 6 g U/l	Total U	± 2.5	± 3.0	-1.1 ± 0.9
B. Isotopic Analyses				
1. Plant Material Assays				
a) Cascade Inventory				
1) Product vs. Grad. Std.	% U^{235}	± 0.20	± 0.02	< 0.1, est.
2) Intermediate vs. Grad. Std.	% U^{235}	± 0.20	± 0.18	< 0.5, est.
3) Waste vs. Syn. Std.	% U^{235}	± 0.20	± 0.17	< 0.2, est.
b) Product				
1) Alpha Counting	% U^{234}	± 1.0	± 0.4	-0.4 ± 0.1
2) Fission Counting	% U^{235}	± 1.0	± 0.4	< 0.2, est.
3) Relative Spectrometer	% U^{234}	± 1.0	± 0.36	< 2.0, est.
4) Relative Spectrometer	% U^{235}	± 0.1	± 0.01	< 0.1, est.
c) Product Stream	% U^{235}	± 0.1	± 0.08	< 0.2, est.
d) Waste	% U^{235}	± 0.3	± 0.28	< 0.2, est.
e) Psi Tests, Pilot Plant	R-1	± 1.5	± 1.5	-1.0 ± 1.5

Analysis		Requested % LEx	Attained % LEx	% Bias
B. Isotopic Analyses (continued)				
2. Miscellaneous Impure Material Assays				
a) Alumina	% U^{235}	± 2.0	± 2.2	< 1.0, est.
b) Carbon	% U^{235}	± 2.0	± 1.6	< 1.0, est.
c) Impure U_3O_8	% U^{235}	± 2.0	± 1.6	0.6 ± 0.6
d) Water Media				
1) 0.07 g U/l	% U^{235}	± 5.0	± 4.5	1.1 ± 1.5
2) 6 g U/l	% U^{235}	± 2.0	± 2.2	0.5 ± 0.6
II. Alpha-Beta Counting				
A. Alpha Counting, Pu traces	α cts/min	± 10	± 37	< 25, est.
B. Beta Counting	Ratio of β cts/min	± 10	± 3	-0.9 ± 0.4
III. Industrial Hygiene				
A. Chemical Analysis				
1. Urine				
a) Uranium	ppb U	Best Readily Obtainable	± 11	-28 ± 13
b) Mercury	ppb Hg	Best Readily Obtainable	± 32	- 5 ± 7
c) Fluorine	ppm F	Best Readily Obtainable	± 46	- 9 ± 17
2. Water	ppb U	Best Readily Obtainable	± 30	-31 ± 6
3. Mud	ppb U	Best Readily Obtainable	± 17	-32 ± 8
B. Counting Analysis				
1. Water				
a) Alpha	α cts/min	± 20	± 30	-28 ± 7
b) Beta	β cts/ min	± 50	± 21	8 ± 6
2. Air	α cts/min	± 10	± 7.2	No Estimate

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Analysis		Requested % LE_x	Attained % LE_x	% Bias
IV. Specification Analysis of Material Other Than Uranium				
A. Oil**				
1. Flash Point	°F	ASTM*	± 9°F	No Estimate
2. Fire Point	°F	ASTM	±24°F	No Estimate
3. Pour Point	°F	ASTM	±18°F	No Estimate
4. Viscosity				
a) at 100°F	sec	ASTM	± 2 sec	No Estimate
b) at 130°F	sec	ASTM	± 2 sec	No Estimate
c) at 210°F	sec	ASTM	± 1 sec	No Estimate
B. Nickel Powder**				
C. Steel				
1. Sulfur	% S	± 5	± 25	12 ± 15
2. Carbon				
a) > 0.1% C	% C	Best Obtainable	± 5.3	0.5 ± 1.8
b) < 0.1% C	% C	Best Obtainable	± 18	3.4 ± 5.7

* ASTM precisions refer to the agreement of duplicate aliquots on the same sample and therefore are not comparable with the "between samples" precisions quoted as the attained LE_x.

** The attained LE_x's for these analyses are quoted in absolute values rather than in per cent.

*** These analyses are run spectrographically and the +100 and -50% LE from spectrographic error is not included in these figures.

Analysis		Requested % LEx	Attained % LE_x	% Bias
V. Barrier Tests*				
A. 6" Tubes (Series Tester)				
1. Slope Factor		Best Obtainable		No Estimate
2. Porosity		Best Obtainable		No Estimate
B. 3' Tubes (Bridge Tester)				
1. Slope Factor		Best Obtainable		No Estimate
2. Porosity		Best Obtainable		No Estimate
C. 7' and 9' Tubes (Series Tester)				
1. Slope Factor		Best Obtainable		No Estimate
2. Porosity		Best Obtainable		No Estimate
3. Porosity		Best Obtainable		No Estimate
D. 7' and 9' Tubes (Bridge Tester)				
1. Slope Factor		Best Obtainable		No Estimate
2. Porosity		Best Obtainable		No Estimate
VI. Other Analyses				
A. UF_4				
1. Uranium	% U	Best Obtainable	± 0.32	< 0.4, est.
2. Fluoride	% F	Best Obtainable	± 0.91	< 1.0, est.
3. Tetra Assay	% U^{+4}	Best Obtainable	± 0.56	< 0.6, est.

* The attained LE_x's for these analyses are quoted in absolute values rather than per cent.

VII. Laboratory Differences — Difference in Means*

A. Feed Samples

1. Per cent free UO_2 in UO_2

a) K-25 minus Mallinckrodt	-0.33 ± 0.10
b) K-25 minus New Brunswick	-0.31 ± 0.14
c) K-25 minus Harshaw	-0.35 ± 0.13

2. Per cent UF_4 in UF_4

a) K-25 minus Mallinckrodt	0.03 ± 0.12
b) K-25 minus New Brunswick	-0.20 ± 0.13
c) K-25 minus Harshaw	-0.09 ± 0.16

B. Product Samples (K-25 minus Y-12)

1. Chemical Analysis (% Difference)

a) Uncorrected analysis**	0.06 ± 0.01
b) Net Weight of Samples	-0.01 ± 0.01
c) Total Uranium Received at K-25	
1) By uncorrected analysis **	0.04 ± 0.02
2) By corrected analysis**	0.08 ± 0.04

2. Isotopic Analysis

a) Product Cylinders	
1) % U-235	-0.050±0.012
2) % U-234	0.025±0.004
b) Controls	
1) % U-235	-0.053± 0.012
2) % U-234	0.021± 0.005

C. Plant Streams (Field Lab. minus Lab. C.)

1. Product % U-235	-0.006± 0.009
2. Waste % U-235	-0.001± 0.003

D. Barrier Material (Barrier Testing minus Barrier Plant)

* The difference between the means and the LE on that difference have been quoted over the entire year and are quoted in absolute values.

** Corrections mentioned are for estimated positive biases of 0.09% at Y-12 and 0.05%at K

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